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CHEMICAL EXPERIMENTS

WARD

FIFTH EDITION



Name



HANDBOOK
OF
CHEMICAL EXPERIMENTS
FOR
DENTAL STUDENTS

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FIFTH EDITION



NEW YORK
D. VAN NOSTRAND COMPANY
8 WARREN STREET
1921

QD45
W3
1921

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THE NEW ERA PRINTING COMPANY
LANCASTER, PA.

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APR -5 1921

FOREWORD

In compiling this laboratory handbook the author has merely put into book form the general order of instruction and experiments used in his laboratory for a number of years. His aim has been to lead the student to do as much theoretical and practical work by himself as possible. All questions asked should be answered, formulæ inserted for all substances, and chemical equations worked out.

It is the sincere wish of the author that this laboratory guide may help in the broadening and deepening of chemical knowledge and tend to promote the student's appreciation of the importance of this subject in the study of dental medicine.

DE L. W. W.

NEW YORK, December 1, 1916.

PREFACE TO THE FIFTH EDITION

Exhaustion of the fourth edition has required a fifth for the laboratory work of this year. Revision and corrections have been made to bring the work up to date.

DE L. W. W.

NEW YORK, January, 1921.

GENERAL CHEMISTRY

THE BUNSEN BURNER

This is a special form of burner for producing heat without light and smoke, by the more complete combustion of illuminating gas. Examine burner and briefly describe it.

The gas and air mix in the lower part of the tube and burn at the top. As the gas and the air are intimately mixed combustion takes place in all parts of the flame. Now close the valve at the bottom and notice the luminous flame. Pass a toothpick slowly through this flame. Then open the air valve and notice the difference in the color of the flame; also pass a toothpick slowly through the flame. Explain why the flame is almost colorless at first, and yellow afterwards (when the air valve is closed). What difference do you notice between the two toothpicks? Explain. There are various modifications of the Bunsen burner, but the principle is the same in all.

BLOWPIPE

The simple blowpipe is an instrument for producing a higher temperature than can be obtained with an ordinary flame, by introducing air (oxygen) into a flame and thus producing more complete combustion.

It consists of a brass tube about 12 inches long. The smaller end is bent at a right angle, and terminates in a reinforced opening. At the larger end a rubber mouthpiece is

usually employed to prevent contact of the brass with the lips, and also facilitate the operation of blowing.

By placing the tip of the blowpipe at the edge of the flame, and blowing strongly a pointed flame, colorless at the outer part (oxidizing) yellowish within (reducing) is formed. Substances heated at the first part tend to oxidize; oxides heated in the second part tend to be reduced.

A yellowish flame rich in carbon or carbon gases is reducing.

A bluish or colorless flame, due to complete combustion, is oxidizing.

The proper and continuous use of the blowpipe (blowing a steady flame for two or three minutes) requires care and practice. The student must first learn to breathe regularly with the cheeks distended. In this way a continuous current of air can be supplied to the blowpipe. The cheek muscles must be exercised so as to keep up a continual tension on the air in the mouth. The lungs act like a force pump, and the mouth with the cheek muscles act like an air chamber. (See force pumps in physics.)

THE WORKING OF GLASS TUBES, RODS, ETC.

The working of glass tubes, etc., into various forms, pieces of apparatus, and so forth, is treated in many books on elementary chemistry, also in special works on glass blowing. It is generally sufficient to be able to cut a piece of tubing, to round the ends by holding in a flame until soft, closing one end of the tube, and then blowing a bulb. Also bending and drawing out glass tubing and rods.

In order to break off a piece of glass rod or tube, make a small scratch with a triangular file. Then grasp the tube or rod with both hands, the thumbs extending along the tube and

almost touching, and the scratch being on the opposite side. Now press gently with the thumbs and pull at the same time, when the tube will break at the mark.

Before going further, the sharp edges of the tube or rod should be rounded, by holding the glass in the Bunsen flame, turning constantly, until the edges have melted and become round.

If the above is continued, with a tube, the opening will become smaller and smaller and finally close.

The closed tube can be used for making a bulb by heating about one-half an inch of the closed end of the tube and turning constantly. When quite hot and soft, remove from the flame, apply the lips to the open end and blow slowly, turning the tube all the time. The soft glass will expand and form a bulb.

Tubes may be drawn out into points by placing the "wing top" on the burner (which gives a long, flat flame), and then holding the tube in the flame, and turning. When the tube is soft, pull slowly, turning at the same time, and the soft tube will be drawn out to a smaller diameter. If the same method of heating a tube is used, when the tube is soft, it may be bent at any angle.

Melting Point Tube.

Take a piece of medium glass tubing $\frac{3}{8}$ -inch bore and about 6 inches long. Heat it in the flame and when soft draw it down to a tube about $\frac{1}{8}$ inch. Cut off a piece of the reduced tube, about 3 inches long and seal one end by heating in the flame until closed. Then heat the tube carefully about 1 inch from the closed end and when soft reduce the caliber or make a constriction by pulling slightly.

For further information consult Thomas Bolas, "Glass Blowing and Glass Working."

WASH BOTTLE

Examine the wash bottle and note each tube and its arrangement. When the wash bottle, filled with water, is inverted, the water will flow out of the tube with the larger opening. When the bottle is held in an upright position, and a current of air forced through the large tube by applying the lips and blowing, a fine jet of water will be forced out through the smaller tube.

SOME IMPORTANT OPERATIONS

Solution.—The diffusion of a solid, liquid, or a gas, through a liquid. It may be either mechanical, physical, or chemical.

Sedimentation.—The separation of a mixture of a finely divided solid and a liquid, by letting it stand, the solid will fall to the bottom of the vessel by gravity, *e. g.*, muddy water.

Decantation.—The pouring off of the clear liquid after sedimentation.

Filtration.—The separation of a mixture of a solid and a liquid (muddy water) by means of a porous substance or filter, upon which the mixture is usually poured. The liquid passes through (filtrate), but the solid (residue) is retained on the filter. Glass funnels with discs of paper made into cones and placed in the funnel are generally used in chemical operations.

Precipitation.—The separating out of a substance, in the solid form, from a solution. It may be either physical or chemical. The former when the solution changes, as the separation of a salt from an aqueous solution by the addition of

alcohol. The latter, the formation of a new substance, insoluble in the solvent, by changing the composition of the substance in solution; *as* washing soda added to lime water, forms calcium carbonate, which is insoluble.

Evaporation.—The separation of a liquid from a solid, either in suspension or solution, by converting the liquid into vapor and allowing it to escape.

Distillation.—It is the same as evaporation except that the vapor is not allowed to escape, but is condensed by cooling, and collected.

Sublimation.—The vaporization of a solid and its condensation back to the solid state.

Solubility.—(Unless the solvent is mentioned, water will be used.)

The solubility of a substance may be determined by placing a small amount in a test tube, adding distilled water and shaking. If the substance disappears it is soluble. It may be only partially or slightly soluble and appear not to dissolve. In that case filter off the liquid and evaporate the filtrate in a porcelain dish. If the substance was soluble there will be a residue left in the dish.

GENERAL EXAMINATION OF SUBSTANCES

As many students, in beginning the study of chemistry, and the examination of the different forms of matter, are at a loss as to the method of procedure, the scheme given below was introduced into my laboratory a number of years ago.

This scheme should be followed for each substance and answers given on the blank pages.

Solids.

Color :

Odor :

Crystalline or amorphous, examine particles under microscope. Draw at least five different forms.

Hardness :

Scratched with the nail—soft.

Scratched with a file—hard.

Not scratched with a file—very hard.

Melting-point : (see page 7).

Specific gravity :

Solubility :

Magnetic : usually for metals only.

Action of heat : Charring indicates a carbon compound.

Combustible.

DETERMINATION OF MELTING-POINT

Prepare a melting-point tube. A small thin glass tube about 2 to 3 inches long, bore $\frac{1}{16}$ inch and closed at one end. About $\frac{1}{2}$ inch from the closed end make a slight constriction by heating and drawing out. Fasten the tube to a thermometer, by means of a rubber band, so that the constriction is opposite the bulb. Bore a hole in a cork; insert the thermometer. Place a small piece of the substance whose melting-point is to be determined, in the open end of the small tube. It should be large enough to be retained at the constriction. The thermometer is immersed in melted paraffine contained in a test tube, so that the bulb is about 2 inches from the bottom of the tube. Heat the paraffine with a small flame and note the temperature at which the substance softens or slips down the construction which will be its melting-point.

Liquids.

Color :

Odor :

Viscosity :

Specific gravity :

Boiling-point :

Action on litmus paper :

Blue becomes red = acid action.

Red becomes blue = alkaline action.

Neither affected = neutral.

Both slightly = amphoteric.

Combustible :

Is it a solution or a simple liquid? Evaporate some and look for a residue.

Gases.

As gases tend to escape, unless confined, it is necessary to keep them in closed vessels, and also to prevent their mixing with the air. They also require special methods for collection, depending upon the nature of the gas.

If heavier than air they may be collected by downward displacement, that is, by allowing them to flow into an upright bottle and displace the air (like a liquid). If lighter than air the bottle is inverted, and the gas, rising in the bottle, pushes out the air (upward displacement). Gases may also be collected over water (the method most commonly used), which is employed when the gas is only slightly soluble in water or not affected chemically by it. The bottle is filled with water, a glass plate placed on the mouth, and pressed down with the finger. This system is then inverted and the neck of the bottle carried below the surface of the water contained in a pan or

vessel called a pneumatic trough. The plate is then removed, the water remaining in the bottle. The gas is then allowed to enter at the mouth of the bottle, and rising displaces the water. When the bottle is full the glass plate is again placed on the mouth and the system removed from the trough. In some cases water cannot be used, and then a mercury trough is substituted.

Color:

Odor:

Taste:

Specific gravity. Whether it is lighter or heavier than air is usually sufficient.

Solubility:

Partly remove the cover, pour some water into the bottle (about 1 inch), replace the cover, and shake. The force required to remove the cover, will give some idea of the quantity of gas dissolved, as the pressure will be proportional to the solubility of the gas.

Action on litmus paper: Dry gases do not act upon dry litmus, the paper must be moistened with water before making the test.

Is it combustible?

Does it support combustion (like air)?

Is it indifferent, neither burns nor supports combustion?

THE STUDY OF CHEMICAL CHANGES

(1) **Analytic** (separation). The separation of a substance into simpler forms of matter (compounds or elements).

(a) Examine the substance given:

Condition: crystalline or amorphous.

Color:

Solubility :

Heat some in a glass bulb, with tube bent in form of a V. Is a gas given off? Test with flame, test with spark on end of a toothpick. Does anything collect in the "V" part of the tube? If so, remove, and see if you can identify it.

Is the change chemical? Give reasons.

(b) Examine limestone or chalk:

Color :

Solubility :

Action on litmus paper :

Heat some in a small, hard glass tube, and test for gases. Examine the residue in the tube. Has it changed in appearance? Does it dissolve in water? Has it any action on litmus? Is the change chemical? Give reasons.

(2) **Synthetic** (putting together).

Examine iron filings.

Examine flowers of sulphur.

Prepare a mixture of the two in about equal parts, rub well together and answer the following:

Are the iron and sulphur separate? Use microscope.

Will a magnet separate the iron from the sulphur?

Will carbon tetrachloride dissolve one and leave the other?

Will hydrochloric acid dissolve one and leave the other?

If a gas is given off, notice the odor.

Heat some of the mixture in a small test tube. Does it glow?

Then cool, remove the mass from the tube and examine:

Microscope.

Magnet.

Carbon tetrachloride.

Hydrochloric acid, and note if the gas given off has the same odor as before.

Draw conclusions, as to a chemical change.

Other chemical changes.—(a) Examine a piece of zinc, then place it in a large test tube containing about 2 inches of hydrochloric acid dilute.

Is a gas given off? It so, test.

When all the zinc has disappeared filter the solution, if necessary, and evaporate the filtrate in a porcelain dish.

Examine the residue, and give reasons for thinking a chemical change has taken place.

Allow the dry residue to stand until the next period. What change do you observe?

(b) Examine some crystals of copper sulphate. Heat some in a tube. What do you observe?

Dissolve some of the crystals in water, note color of the solution.

Place a piece of clean iron into the solution, warm, allow to stand for a few minutes and answer the following:

Has the solution changed in color?

Remove some of the red substance formed. Dry and rub with a knife blade. Can you identify it?

Explain the changes.

PREPARATION AND PROPERTIES OF OXYGEN

Required:

Hard glass tube, side-neck, and cork.

Wash bottle for gas, with rubber stopper and tubes.

Tin pan, glass bottles and flat covers, for collecting gas over water. . Oxygen mixture (potassium chlorate two parts, and manganese dioxide one part).

Sodium hydroxide solution for wash bottle.

Set up apparatus as shown on the demonstrating table, heat

the oxygen mixture and when the gas appears at the delivery tube (spark on end of tooth pick will burst into flame), collect four bottles and make the following tests:

(1) With one bottle determine:

Color:

Odor:

Solubility:

Action on litmus:

(2) In another bottle burn some wood, add water, shake and test with litmus paper.

(3) Burn sulphur in another and test as in (2).

(4) Burn magnesium ribbon in another and test as in (2).

What are your conclusions in regard to combustion in oxygen?

FLAME TESTS

Some substances, when heated in the clear Bunsen flame, will impart a color which is characteristic for that substance and serves for its identification. These tests are made by heating the substance on a clean platinum wire, as follows:

Clean the platinum wire by dipping it into concentrated hydrochloric acid and holding it in the flame, until all color has disappeared. Then, without touching the wire, dip it into the substance to be tested and hold in the flame again; note color imparted. If to the substance (being a solid) a small amount of pure hydrochloric acid concentrated, is added, better results will be obtained, as the chlorides formed are very volatile.

Report flame color of the following salts, cleaning the wire before each test:

Potassium chloride.

Calcium chloride.

Lithium chloride.

Barium chloride.

Strontium chloride.

Sodium chloride.

Instead of platinum wires thin charcoal sticks may be used. Heat the stick in the flame to be sure that it is free from impurities, then dip it into the solution or the solid substance, hold it in the flame and note the color.

HYDROGEN

Set up an apparatus as shown on demonstrating table; the large test tube, supported in the iron clamp, with rubber stopper and delivery tube. Fill the large test tube about one-third full with sulphuric acid dilute and add a few pieces of zinc. As soon as there is a brisk evolution of the gas* place a small test tube over the delivery tube. In a few minutes remove the small test tube, keeping the mouth downward, and test the gas with flame; when the gas burns quietly or without explosion fill several test tubes in the same manner and make the usual tests.

The above apparatus can be used for other light gases.

Color:

Odor. (May be an odor from impure zinc.)

Action on litmus paper.

Does the gas support combustion?

Collect some in a dry tube, and ignite what is formed.

Give reasons for your answers.

Hydrogen can also be prepared (in a pure state) by the action of sodium on water

* If the gas is evolved very slowly, add a few drops of a solution of copper sulphate to the mixture; can you explain how this accelerates the reaction?

Fill a test tube with water, place thumb over end and invert in a basin of water. Take a small piece of sodium, in the iron forces, bring it quickly below the mouth of the test tube and release. What do you observe? When the action is over test the gas. Also test the water in the basin with red litmus paper. Explain the reaction.

Hydrogen is the essential element of all acids, but hydrogen ions only give an acid action; the element is neutral.

SOME ACID TESTS

The so-called acid tests are of considerable importance, and have to be made from time to time even in the early study of chemistry. Two tests with method will therefore be given.

These are made for the part or radicle associated with the hydrogen (anions).

Test for chlorine ions.

Make the solution slightly acid with dilute nitric acid, then add a few drops of silver nitrate solution. A white, curdy precipitate is formed, which blackens in the light, and is soluble in ammonium hydroxide.

Test for sulphuric ions.

Make a solution slightly acid with dilute hydrochloric acid and add a few drops of barium chloride solution, a heavy white precipitate of barium sulphate is formed. Other acid tests will be given later.

HYDROGEN DIOXIDE

Pour about 20 c.c. of dilute sulphuric acid into a small breaker, place on a wire gauze on the ring stand. Add, in small portions at a time, 10 grams of powdered barium dioxide and stir well. Heat the mixture carefully for a few minutes,

but do not boil. Then add barium carbonate slowly with stirring until there is no further effervescence, showing that excess of sulphuric acid has been neutralized. Heat for a short time, filter off the barium sulphate, cool the filtrate and test with potassium iodide starch mixture. A blue coloring of the starch indicates hydrogen peroxide.

Make the following tests:

(1) Add some dilute chromic acid solution to hydrogen peroxide. Note change of color due to the formation of perchromic acid.

(2) Add some hydrogen peroxide to a dilute solution of potassium permanganate. Observe change and explain the reaction.

(3) Hydrogen peroxide added to a solution of titanous sulphate $\text{Ti}(\text{SO}_4)_2$ gives a yellow color due to the formation of pertitanic acid.

EXAMINATION OF THE HALOGENS

Fluorine.—This element has been prepared only with great difficulty, so will not be studied in the laboratory.

Hydrofluoric Acid.—(1) Place some calcium fluoride in a leaden dish and add concentrated sulphuric acid sufficient to cover the powder. Place on a ring stand and heat with a small flame. Cover the convex side of a watch glass with paraffine, by warming the glass and rubbing a piece of paraffine over the surface. When cool make some marks with a hard instrument on the paraffine so as to expose the glass. Place the glass, convex side down, on the lead dish and let it remain for half an hour. Then take off the watch glass, and remove the paraffine by warming and rubbing with a towel. Explain the phenomenon and write the equations.

(2) Clean a microscopic slide and make some tracings with hydrofluoric acid solution. Allow to stand for ten minutes, wash off with water and explain the phenomenon.

(3) Clean a piece of glazed porcelain, a portion of the outside of a porcelain dish will do; then place a drop or two of a solution of hydrofluoric acid on the dish, allow to remain thus for a few minutes. Wipe off the acid with a wet cloth, and note the roughness of the surface of the dish. If the result is not good the first time repeat the application of the acid to the same two or three times. Explain.

Chlorine.—Place about half an inch of manganese dioxide in a large test tube, add about 10 c.c. of hydrochloric acid conc., heat carefully, and collect the gas by downward displacement.

Color:

Odor:

Light or heavy: Can the gas be poured from one tube to another?

Solubility: allow the gas to come in contact with some water in another tube, and shake well.

Action on litmus paper:

Does it burn?

Does it support combustion?

Try action on a piece of dyed cotton cloth: the cloth must be wet. Why?

Introduce some bleaching powder into a large test tube, add some sulphuric acid dilute; warm and try the action on litmus paper, first dry, then wet. Explain.

Hydrochloric Acid Gas.—Place some sodium chloride in a large test tube, fitted with rubber stopper and doubly bent tube. Add sufficient concentrated sulphuric acid to cover the salt

completely, insert the stopper and heat carefully. Write the equation.

Test with litmus paper, first dry, then wet.

Fill several tubes and test gas as usual.

Make a solution and test:

Hydrogen ions.

Chlorine ions.

Aqua Regia.—A mixture of hydrochloric and nitric acids, which gives off chlorine. Write the equation, and explain.

Add a few pieces of gold to a mixture of dilute hydrochloric acid (3 parts) and dilute nitric acid (1 part), heat until gold is dissolved. Write the equation.

Bromine.—Prepare a mixture of potassium bromide and manganese dioxide.

Place in a large test tube same apparatus as for hydrochloric acid and add some dilute sulphuric acid.

What is set free?

Color:

Odor:

Light or heavy?

Is it soluble in water? In carbon tetrachloride?

Add a few drops of dilute hydrochloric acid to a bromide solution and about 1 c.c. of carbon tetrachloride, which will fall to the bottom of the tube. Then add some chlorine water, shake, let stand and observe that the globule at the bottom of the tube is colored yellow or yellow-red, due to the presence of bromine.

Add more chlorine water and see if the color can be bleached.

Iodine.—Condition:

Form:

Color :

Odor :

Solubility in :

Water.

Alcohol.

Carbon tetrachloride.

Note colors of the last two solutions. Can you explain the difference?

The tincture contains potassium iodide. Why?

Heat some iodine in a small test tube held almost horizontal and observe.

Does it melt?

Does it vaporize without melting (sublime)?

What is the color of the vapor?

Condense same and examine particles under microscope.

Test for Iodine.—Add one drop of Lugol's solution* to some dilute starch paste in a test tube, shake and note color.

Heat the tube and note what happens.

Allow to cool and note again.

Can you explain the changes?

Test for Iodine Ions.—This test is made in the same manner as that for bromine (see page 17).

Add more chlorine water and see if the color disappears.

Iodine and bromine ions precipitate silver nitrate solution as well as chlorine ions giving silver iodide and bromide, respectively. All are affected by light. (Photography.)

NITROGEN

Preparation : by burning out the oxygen from air.

Tall, graduated bell jar, open at the top; deflagrating spoon

* A solution of potassium iodide and iodine in water.

for burning phosphorus and rubber stopper ; also a deep, wide glass jar or pneumatic trough, containing some water.

Place the bell jar partly in the water, read the volume of air, and, without changing the level of the water, introduce the deflagrating spoon with some burning phosphorus, also inserting the rubber stopper. Lower the jar somewhat to prevent the escape of any air and when the combustion ceases allow the gas to cool and the white vapor to be absorbed by the water. Then raise or lower the jar until the level of the water is the same within and without and read the volume of the gas. Why should the water levels be made the same?

Determine the per cent of this gas in air?

Does it burn?

Does it support combustion?

Has it any effect on lime water?

It is not pure nitrogen, but contains about 1 per cent of argon and small quantities of rare gases.

Ammonia

Heat a mixture of an ammonium salt, with calcium hydroxide using an apparatus like that for hydrogen, and examine the gas evolved.

Color:

Odor: (Smell carefully.)

Action on wet, red litmus paper:

Solubility.

Does it burn?

Does it support combustion?

Moisten a glass rod with concentrated hydrochloric acid, and hold in the gas; what do you observe? Explain.

Pass some of the gas into water and test the solution. What is formed? and why does the water become alkaline?

Test for ammonium, either in a salt or in solution (ammonium ions). Place some of the substance in a test tube add about one inch of caustic soda solution, heat and test gas with wet, red litmus paper. The paper will become blue if ammonium is present.

Nessler's Test.—Nessler's solution* added to a colorless solution of an ammonium compound will give a yellow color or precipitate, depending upon the amount present. This is a very delicate test so use a very weak solution. (1 : 100,000.)

Nitric Oxide

Prepare a gas generator consisting of a wide-mouthed bottle, rubber stopper with two holes, funnel and delivery tube; also pneumatic trough.

Place some copper in the generator with sufficient water to cover it. Then pour concentrated nitric acid through the funnel tube until there is a brisk evolution of gas. Collect three bottles; the gas should be colorless. The orange fumes appearing in the generator are nitrogen dioxide and are dissolved by the water.

Open one bottle of gas and notice change of color. Explain. Try the combustion of sulphur.

Try the combustion of phosphorus. Explain the difference.

Nitric Acid

Set up a glass-stoppered retort and receiver. The retort supported on a wire gauze and held by a clamp. All the glass apparatus should be dry.

Introduce some sodium nitrate into the retort, and add

* A solution of mercuric iodide in potassium iodide, and made alkaline with caustic soda.

enough concentrated sulphuric acid to cover it. Heat carefully and distil off the nitric acid. Make the following tests:

Color:

Odor:

Solubility:

Taste. Place one drop in a test tube half full of water, mix and taste.

Action on litmus paper.

Action on copper: write the equation.

Action on tin: and state if it differs from that of copper.

Action on wool: Place a small mass of worsted in the neck of a test tube containing about half an inch of concentrated nitric acid. Heat, record phenomena.

Action on turpentine, an inflammable substance. Place some nitric acid in a test tube, standing in the rack, then add some turpentine, a few drops at a time, by means of a long glass tube. (Do not stand near the test tube.) Note phenomena and explain.

Test for nitric ions.

Add a crystal of ferrous sulphate to the solution, then allow concentrated sulphuric acid to flow down the side of the tube slowly and collect in the bottom, where the two liquids meet, a brown ring will appear.

Preparation and Properties of Nitrous Oxide

Set up an apparatus, as shown, on the demonstrating table. Hard glass tube, with side neck and a cork. The tube supported in a clamp, and the side neck joined to two wash bottles. The first contains a solution of ferrous sulphate; the second a solution of caustic soda. The delivery tube extends into warm water in the pneumatic trough. Place some ammonium nitrate

in the hard glass tube. Insert the cork into the neck and heat carefully. When the salt is melted and appears to begin to boil, test the gas evolved at the delivery tube with a spark on the end of a toothpick. When the spark is in flame, collect six bottles of the gas. Remove the cork from the hard glass tube before taking away the flame, to prevent back suction of the ferrous sulphate solution, causing an explosion.

Examine the gas:

Color:

Odor:

Taste:

Solubility.

Action on litmus paper.

Does it burn?

Burn wood and test.

Burn sulphur and test.

Burn magnesium and test.

Do you think it would support life?

Test the ammonium nitrate for:

(a) Ammonium group.

(b) Nitrate group.

Difference between nitrous oxide and air (oxygen).

Fill two wide-mouth bottles with nitric oxide, cover carefully, so as to exclude all air (oxygen). Then fill a bottle (with the same sized neck) with nitrous oxide, being also careful to exclude air; also fill one bottle with air (oxygen). Arrange the bottles containing the air and nitrous oxide on the table, mouths upward. Bring the two bottles containing the nitric oxide, mouths downward, over the others; rapidly remove the covers and bring the bottles set mouth to mouth, note difference between the two systems and explain.

EXAMINATION OF CARBON

Heat a piece of wood charcoal on the wire gauze with the Bunsen flame, and explain the change.

Carbon is always obtained when organic matter (plant or animal), is heated in closed vessels or out of contact with air. Gaseous and liquid products are also formed.

Destructive distillation. Heat a piece of wood in a large test tube, held almost horizontal. Explain changes and identify products formed as far as possible.

Carbon Dioxide

Prepare a gas generator, consisting of a wide-mouth bottle, rubber stopper with two holes, funnel tube, delivery tube, pneumatic trough and bottles for collecting the gas.

Introduce some limestone into the generator, with some water to cover it. Then add enough hydrochloric acid to start a brisk effervescence. Test for the gas at the delivery tube with a spark on the end of a toothpick. When the spark is extinguished collect the gas.

Then make the following tests:

(1) Collect a bottle of gas by downward displacement (dry gas), and test with blue litmus paper, first dry then wet; note difference and explain.

(2) Burn some magnesium ribbon in a bottle of the gas; can you explain the combustion?

(3) Add some lime water to a bottle of the gas, shake and explain.

(4) Pass the gas into lime water, note action as in (3), continue to pass in the gas until clear. Can you explain the change?

Divide the solution from (4) into two parts:

(5) To one portion add some tincture of soap solution, shake and explain.

(6) Boil the other portion and explain.

(7) Filter the mixture obtained from (6), add soap solution, shake and explain the difference between (5) and (7).

(8) Breathe into lime water contained in a large test tube. What do you observe? Explain.

Carbon dioxide is formed in all cases where carbon is oxidized by excess of oxygen.

Carbon Monoxide*

This gas is formed by the reduction of carbon dioxide, also by the decomposition of certain carbon compounds.

Heat oxalic acid with concentrated sulphuric acid in the tube of an apparatus similar to that for hydrogen and pass the gases evolved through a solution of caustic soda to remove the carbon dioxide, also formed. Collect the gas over water.

Test its properties as usual.

Burn some in a bottle with the mouth downward, and test with lime water. Explain.

Methane (Marsh Gas)

Heat a mixture of soda-lime and dry sodium acetate in a large test tube, etc., collect the gas over water.

Color.

Odor.

Light or heavy?

Solubility.

Action on litmus.

Does it burn?

* This gas is very poisonous, so do not inhale any.

Does it support combustion?

Burn some and test products with litmus paper, with lime water.

Acetylene*

Pour about 10 c.c. of water into the large test tube of the hydrogen generator and add two or three small pieces of calcium carbide; notice the effervescence. Collect the gas over water.

Color.

Odor.

Light or heavy?

Action on litmus.

Does it support combustion?

Burn gas and notice the character of the flame, test the products of combustion with lime water.

Cyanogen†

Place some mercuric cyanide in a test tube, fitted with a cork and a jet tube. Heat in the Bunsen flame, and note the burning gas.

Explain the color of the flame and products of combustion.

SULPHUR

Color.

Odor.

Taste.

Solubility, in water, carbon disulphide, ether.

Prepare a solution of sulphur in carbon tetrachloride, filter

* This gas is explosive and also poisonous.

† This gas is exceedingly poisonous, so do not inhale any.

and allow some of the filtrate to fall on a microscopic slide; when the solvent has evaporated examine the crystals under the microscope, draw some and try and determine their crystalline form.

Sulphur Dioxide

Place some sodium sulphite in a large test tube, fitted with rubber stopper and bent delivery tube, add some hydrochloric acid and collect the gas by downward displacement.

Color.

Odor.

Action on litmus, first dry, then wet.

Solubility.

Does it burn?

Try action on some weak fuchsin solution and explain; does any change take place after standing several hours? Explain.

Sulphuric Acid

Bring two bottles together mouth to mouth; the one above containing sulphur dioxide, the one below nitrogen dioxide, allow them to stand and observe changes. Later, add a small amount of water to each, shake, mix and test for: (a) hydrogen ions, (b) sulphuric ions with barium chloride solution a white precipitate of BaSO_4 is formed. Write all the equations.

Sulphuric acid may be prepared in a small way for tests, by the following method:*

Heat a mixture of sulphur with about twice its bulk of potassium chlorate in a small test tube, *use a test tube holder*.

* This laboratory method of preparing sulphuric acid was first suggested to me a number of years ago by Prof. H. T. Vulté, Teachers College, Columbia University.

Explain the phenomenon (deflagration). Let the tube cool, add some distilled water, shake and let stand until liquid is clear. Pour off the clear liquid and test.

(1) For hydrogen ions.

(2) For sulphuric ions.

Write all the equations.

Examine some of the sulphuric acid in the bottle on the deck for :

Color.

Odor.

Viscosity (limpid or oily).

Pour some into cold water very carefully. Note effect and explain.*

Try the action on :

(1) Zinc ; first dilute, then concentrated.

(2) Copper ; first dilute, then concentrated, with heat.

(3) Sugar ; place some sugar in a large test tube, supported in a holder, then add some concentrated sulphuric acid carefully.

(4) Wool ; make test the same as (3). Explain each action.

Normal Solutions.

Explain what is meant by normal sulphuric acid, normal hydrochloric acid and normal sodium hydroxide solutions.

Prepare 50 c.c. of normal sulphuric acid, starting with 50 per cent sulphuric acid. Use a burette for measuring the acid and the distilled water. Determination of the strength of an unknown sodium hydroxide solution. Place 20 c.c. of the solution in a small beaker, add a few drops of litmus solution

* When mixing the acid with water the above *order* should be used to avoid serious accidents, which are not uncommon.

and note the blue color due to the alkali or hydroxyl ions. Fill a burette to the 0 mark with the N-sulphuric acid and support it in the iron clamp on the ring stand. Allow the acid to fall drop by drop into the alkali, stirring the mixture with a glass rod, until the blue color becomes pink or just red, showing that the alkali has been neutralized. Read off the number of c.c. of acid used and calculate the strength of the sodium hydroxide, or the amount of the sodium hydroxide in the 50 c.c. of solution.

Carbon Disulphide

Condition:

Color:

Odor:

Light or heavy?

Solubility:

Is it inflammable?

Solvent action on:

(1) Sulphur.

(2) Rubber.

How would you distinguish this compound from:

(a) Carbon tetrachloride? (b) Chloroform?

Hydrogen Sulphide

Arrange a large test tube with rubber stopper and bent glass tube as in other experiments for the preparation of gases. Introduce some pieces of ferrous sulphide into the tube and add enough dilute hydrochloric acid to cover them. Observe the effervescence. Examine the gas:

Color.

Odor.

Solubility.

Action on litmus paper.

Does it burn? If so, what is found?

Does it support combustion?

Test with lead paper.*

Reactions of Some Metals (Cations) with Hydrogen Sulphide

Treat solutions of the following metals with hydrogen sulphide, note the effect on each, and write the equation.

Zinc.—Precipitated by hydrogen sulphide in alkaline or acetic acid solution as zinc sulphide; white precipitate soluble in dilute mineral acids.

Lead.—Precipitated by hydrogen sulphide in acid solution as lead sulphide, black precipitate, insoluble in dilute acids, is converted into lead sulphate (white), by the action of hydrogen peroxide.

Copper.—Precipitated by hydrogen sulphide in acid solution as copper sulphide, blue black precipitate soluble in nitric acid (distinction from mercuric sulphide).

Cadmium.—Precipitated by hydrogen sulphide in acid solution as cadmium sulphide, bright yellow precipitate soluble in nitric acid, insoluble in ammonium sulphide (distinction from arsenic sesquisulphide).

Bismuth.—Precipitated by hydrogen sulphide in acid solution as bismuth sulphide, black precipitate, soluble in nitric acid (distinction from mercuric sulphide).

Tin.—(Stannous or stannic.)

Both precipitated by hydrogen sulphide in acid solution,

* Made by moistening a piece of filter paper with lead acetate solution.

stannous sulphide, brown precipitate; stannic sulphide, yellow precipitate. Both soluble in ammonium sulphide.

Mercury.—Mercurous: Precipitated by hydrogen sulphide as mercurous sulphide, mixed with mercury, black precipitate, insoluble in ammonium sulphide; soluble in aqua regia.

Mercuric: Precipitated by hydrogen sulphide, in acid solution as mercuric sulphide, black precipitate, insoluble in ammonium sulphide, and dilute nitric acid; soluble in aqua regia.

Silver.—Precipitated by hydrogen sulphide as silver sulphide, black precipitate, soluble in hot nitric acid.

Gold.—Precipitated by hydrogen sulphide in acid solution as gold sulphide, insoluble in acids, soluble in aqua regia, and in ammonium sulphide.

Platinum.—Precipitated by hydrogen sulphide in acid solution as PtS_2 , soluble in ammonium sulphide and aqua regia, insoluble in acids.

BORON

Boric Acid.

Condition:

Color:

Odor:

Taste:

Solubility:

Action on litmus paper:

Make flame test.

Heat some in the loop of the platinum wire until all action ceases. Note the clear bead. What is formed?

Borax.

Color:

Odor :

Taste :

Solubility :

Action on litmus paper :

Make flame test.

Heat with H_2SO_4 conc, add alcohol, ignite and note color of flame.

Borax gives a yellow flame due to the sodium present.

Bead tests.

Make a bead in the loop on the end of the platinum wire, by heating wire and dipping it into the borax and heating again. The bead should be clear and colorless.

Heat bead and dip it into some cupic oxide ; heat again and note color. What is formed ?

Make separate bead tests for the following in the same way and report, color of each, while hot as well as cold.

Cobalt oxide.

Nickel oxide.

Ferric oxide.

Magnanese oxide.

Chromium sesquioxide.

Borax is called an acid flux. Why ?

SILICON

Silica.

Examine silica (clean white sand will do).

Color :

Odor :

Taste :

Solubility :

Fusibility :

Prepare a microcosmic salt (sodium ammonium hydrogen orthophosphate) bead on the platinum wire, when clear, dip bead into silica while hot and heat again; notice the skeleton-like mass floating in the bead while hot which disappears on cooling.

Silicic Acid.

Dissolve some sodium silicate in water, filtering if necessary; observe the character of the solution.

Test with litmus paper.

Make some of the solution acid with hydrochloric, do you observe any change?

Is it still a solution? Can it be filtered?

Dialize some and examine the dialysate.

Evaporate some of the dialized liquid and heat the residue.

Is the residue soluble in water? Make a microscopic salt bead and examine while hot.

PHOSPHORUS

Compare the properties of red and yellow* phosphorus.

Condition:

Color:

Odor:

Solubility in carbon disulphide.

Combustibility with slight heat?

What is formed when exposed to the air?

What is formed when they burn?

Phosphorus Pentoxide.

Place a piece of dry phosphorus in a deflagrating spoon,

* Keep yellow phosphorus under water until wanted. Do not handle it.



heat until it takes fire and allow it to burn in a bottle of dry air.

Examine the product.

Condition.

Color.

Action on water and solubility.

Ortho Phosphoric Acid.

Use the above solution for the following tests:

Hydrogen ions.

Ortho phosphoric ions with:

(a) Ammonium molybdate solution.

(b) Magnesia mixture.*

(c) Silver nitrate.

Examine Meta or Glacial Phosphoric Acid.

Condition:

Color:

Odor:

Solubility:

Action on litmus.

Taste. (Use a dilute solution.)

Use a fresh solution for tests.

Compare Solutions of Each Acid.

Meta.

Orth.

Action on albumen solution.

Action on silver nitrate solution.

Action on barium chloride solution.

Prepare a dental cement by mixing some phosphoric acid solution with zinc oxide. What is formed?

* A solution of magnesium sulphate made alkaline with ammonium hydroxide and ammonium chloride added until the solution is clear.



Phosphorus Acid.

Allow some pieces of yellow phosphorus to stand in a bottle with a small quantity of water. (See the ozone experiment.) Later add a few c.c. of water, shake and pour off the solution from the residual phosphorus. Make the following tests:

(a) Hydrogen ions.

(b) With silver nitrate.

Phosphorus acid may also be made by the action of water on phosphorus trichloride. Write the equation.

Phosphorus Tri-iodide.

Dissolve about 1.0 gram of yellow phosphorus in 25 c.c. of carbon tetrachloride contained in a 200 c.c. flask. Add 12 grams of iodine in small portions at a time, shaking after addition. When all the iodine has been added and passed into combination, connect the flask with a condenser and distil off the carbon tetrachloride, on a water bath. Examine the residue.

Condition.

Color.

Odor.

Melting-point.

Action of water, write the equation.

EXAMINATION OF ARSENIC*

Condition:

Crystalline form:

Color:

* Great care must be taken not to breathe any fumes evolved in these experiments with arsenic, as they are very poisonous.

Is it lustrous?

Is it soft or brittle?

Heat some, mixed with charcoal to prevent oxidation, in a large test tube, held in a horizontal position.

Examine crystals.

Heat some, as above, mixed with potassium nitrate, to oxidize it.

Does it sublime? Examine the crystals.

What is their color?

Remove some of the crystals, dissolve in water and test with litmus paper. What is formed?

Arsenious Oxide.

Condition:

Color:

Odor:

Solubility:

Heat a small piece and note odor; use care.

Test the solution with litmus paper.

Make solution acid with hydrochloric acid dilute and pass in hydrogen sulphide gas. Write the equation.

Place a piece of clear copper into some of the solution, allow to stand for some time, note effect and explain. (Reinsch test.) Remove the copper, wash, dry, and heat carefully with a small flame; note fumes and their odor.

Arsenic Acid.

Condition:

Color:

Odor:

Solubility:

Make a solution slightly acid with hydrochloric acid dilute, and divide into four parts:

(1) Pass in hydrogen sulphide gas and note the effect.

(2) Add a few crystals of sodium sulphite, boil and pass in hydrogen sulphide gas.

Compare (1) and (2) and explain.

(3) Add ammonium molybdate solution, first cold, then warm mixture and note result (dif. H_3PO_4).

(4) Add magnesium mixture;* note ppt. of $\text{NH}_4\text{MgAsO}_4$.

MARSH TEST FOR ARSENIC AND DISTINCTIONS BETWEEN ARSENIC AND ANTIMONY

Hydrogen Arsenide.

Prepare a hydrogen generator consisting of a flask (500 c.c.), rubber stopper with two holes, a funnel tube and a jet for burning the gas. Place some pure zinc in the flask and add enough distilled water to cover it. Then pour in through the funnel tube some pure concentrated sulphuric acid until there is a brisk evolution of gas. Wait a few moments to allow the hydrogen to drive all the air out (otherwise, when a flame is applied there will be an explosion), then light the gas. Observe. The color of the flame: It should be dark blue or colorless, not grayish blue (indicating arsenic).

Place a piece of cold porcelain in the flame. No dark spots should appear on the porcelain. Now add a few drops of a solution of arsenious acid and observe change in the color of the flame.

Place the porcelain in the flame, as before. Brownish black spots with a metallic luster indicate arsenic. Test the

* See foot-note, page 35.

solubility of the spots in: (*a*) chlorine water, should be soluble; (*b*) in hydrochloric acid, should be insoluble.

Hydrogen Antimonide.

Use a similar apparatus and a solution of antimony chloride.

Observe the color of the flame, which should be grayish. The spots on the porcelain should be somewhat sooty in appearance, blacker and lusterless.

Test the solubility of the spots in (*a*) chlorine water, should be insoluble; (*b*) hydrochloric acid, should be soluble.

Other Distinctive Tests.

A solution of arsenious acid or arsenite, made slightly acid with hydrochloric acid, treated with hydrogen sulphide, gives a yellow flocculent precipitate of arsenic sesquisulphide.

With the same reagent a solution of antimony gives an orange flocculent precipitate of antimony sesquisulphide.

QUALITATIVE ANALYSIS

Tests and separations for the more important metals and acids.

For purposes of study and investigation the metals have been placed in groups or classes. The groups being arranged according to the action of certain reagents, which will precipitate some of the metals from a solution and leave others.

Where a partial solution has been made, the mixture is then filtered and by this means a further separation attained. The filtrate is then treated with some other reagent and a still further separation accomplished. These processes are re-

peated until an individual substance is obtained, the latter is then subjected to one or more special tests for its identification.

It is evident from the above that the principles of solution and precipitation are of the utmost importance in qualitative analysis, and should be thoroughly mastered in order to attain good results in this work.

Since a knowledge of the individual tests for each metal must first be acquired, a certain number of tests will be given, and its properties studied; then the metals of that group will be examined together.

The first group is composed of metals whose chlorides are insoluble in water, as lead, silver and mercurous mercury.

METALS

Lead Group (First) Pb, Ag, Hg^I

Solutions of lead salts give with:

Soluble chlorides, a white heavy ppt. of PbCl_2 soluble in boiling water, insoluble in NH_4OH .

Hydrogen sulphide, a black ppt. of PbS , decomposed by hot conc. HNO_3 into PbSO_4 , also converted into PbSO_4 by heating with H_2O_2 .

Soluble sulphates, a heavy white ppt. of PbSO_4 insoluble in dilute acids.

Soluble chromates, a bright yellow ppt. of PbCrO_4 insoluble in acetic acid, soluble in mineral acids.

Solutions of silver salts give with:

Soluble chlorides, a white curdy ppt. of AgCl , which blackens in the light. The ppt. is insoluble in water (hot or cold), soluble in NH_4OH , forming $(\text{NH}_3)_2\text{AgCl}$, which is reprecipitated on the addition of nitric acid dil. The ppt. is soluble in KCN solution.

Hydrogen sulphide a black ppt. of Ag_2S , insoluble in acids, soluble in KCN, sodium thiosulphate and nitric acid.

Soluble iodides and bromides (not fluorides) give heavy curdy ppt. like the chloride; affected by light, also soluble in KCN and sodium thiosulphate.

Soluble chromates a reddish brown ppt. of Ag_2CrO_4 , soluble in strong acids.

Caustic alkalis a yellowish gray ppt. of silver oxide.

Solutions of mercurous salts give with:

Soluble chlorides, a white heavy ppt. of HgCl , insoluble in acids; soluble in aqua regia giving HgCl_2 . Reacts with NH_4OH , forming NH_2HgCl and Hg which is black. Decomposed by fixed alkalis into Hg_2O , also black.

Hydrogen sulphide gives a black ppt. of Hg_2S , insoluble in acids, soluble in aqua regia.

Caustic alkalis a black ppt. Hg_2O , insoluble in excess.

Dry salts, both of mercurous and mercuric mercury, heated with sodium carbonate in a test tube give a gray deposit of mercury (globules) in the cooler part of the tube.

Lead Group Scheme.

Solution containing soluble salts of lead, silver and mercurous mercury.

Add hydrochloric acid dil. in slight excess and filter.

Ppt. A. Filtrate A reject.

Treat with boiling water several times: Residue (a), solution (a), Treat solution (a) with $\text{K}_2\text{Cr}_2\text{O}_7$, a heavy yellow ppt. = PbCrO_4 .

Residue (A). Treat with NH_4OH several times. Residue (B). Solution (b).

To solution (b) add HNO_3 dil., a white curdy ppt. = AgCl .

Residue (B), if black, due to $\text{NH}_2\text{Hg}_2\text{Cl} = \text{Hg}(\text{ous})$.

The second group is made up of two parts. The tin group and the copper group. All these metals are precipitated by hydrogen sulphide in acid solution. The sulphides of the first part are soluble in strong ammonium sulphide, the sulphides of the second part are insoluble in ammonium sulphide.

Copper Group (Second), Hg, Bi, Cu, Cd.

Solution of mercuric salts give with:

Hydrogen sulphide, in excess, in acid solution, a black ppt. of HgS . Insoluble in acids and ammonium sulphide; soluble in aqua regia.

Fixed alkalis, a yellowish-red ppt. of HgO , insoluble in excess.

Ammonium hydroxide, a white ppt. of ammonio-mercuric chloride, NH_2HgCl , insoluble in excess, but soluble in acids.

Potassium iodide, a bright red ppt. of HgI_2 , soluble in excess, forming $(\text{KI})_2\text{HgI}_2$.

Stannous chloride, SnCl_2 , gives a white ppt. at first, of HgCl , with excess a gray or black ppt. of Hg .

Copper, a gray ppt. on the copper, which when heated in a glass tube gives a deposit of mercury globules in the cooler portion of the tube.

Solutions of bismuth salts give with:

Hydrogen sulphide, a black ppt. of Bi_2S_3 , insoluble in dilute acids, soluble in hot HNO_3 .

Fixed alkalis and ammonium hydroxide give a white flocculent ppt. of $\text{Bi}(\text{OH})_3$, insoluble in excess, soluble in dilute acids.

Soluble chromates, a yellow ppt. of basic bismuth chromate soluble in strong acids.

Water, a heavy white ppt. of a basic salt. The nitrate gives BiONO_3 , the chloride gives BiOCl , soluble in nitric or hydrochloric acid.

Potassium stannite (K_2SnO_2),* a black ppt. of Bi.

Salts heated on charcoal with bismuth flux (a mixture of KI and S) give a bright red incrustation.

Solutions of copper salts give with:

Fixed alkali hydroxides, a blue ppt. of $\text{Cu}(\text{OH})_2$, insoluble in excess, soluble in certain organic acids and NH_4OH .

Ammonium hydroxide, at first a blue ppt., soluble in excess forming a complicated salt, having a deep blue color.

Hydrogen sulphide in acid solution, a black ppt. of CuS insoluble in dilute acids except nitric acid (distinct from HgS), insoluble in boiling dilute H_2SO_4 (distinct from CdS).

Potassium cyanide, a brown ppt. of $\text{Cu}(\text{CN})_2$, soluble in excess forming a double cyanide. The latter solution is not precipitated by H_2S (distinction from Cd).

Potassium ferrocyanide [$\text{K}_4\text{Fe}(\text{CN})_6$] in acetic acid, a red brown ppt. of $\text{Cu}_2\text{Fe}(\text{CN})_6$, which will form even in a very dilute solution.

Copper salts heated on charcoal with tin and borax give a red bead due to Cu_2O .

Heated in the borax bead, oxidizing flame, give a green bead hot, and blue when cold.

Solutions of cadmium salts give with:

Fixed alkali hydroxides, a white ppt. of $\text{Cd}(\text{OH})_2$ insoluble in excess.

Ammonium hydroxide, a white ppt., soluble in excess (distinct from Bi).

* Made by adding KOH to SnCl_2 until the ppt. first formed is re-dissolved.

Hydrogen sulphide in acid solution, a bright yellow ppt. of CdS , soluble in boiling dilute sulphuric acid, insoluble in potassium cyanide (distinctions from Cu).

Heated on charcoal in the oxidizing flame gives a brown coat of the oxide.

The borax bead is yellow when hot, colorless when cold.

Copper Group Scheme.

If the solution is not already acid make it slightly acid with HNO_3 dilute and pass in H_2S gas in excess and filter.

Ppt. A. Filter reject.

Treat precipitate (A) with HNO_3 dilute, several times solution (A).

Residue $\text{A} = \text{HgS}$: Dissolve in aqua regia, boil out Cl , and filter if necessary. Add SnCl_2 , a white ppt. becoming black on further addition of $\text{SnCl}_2 = \text{Hg}$.

Solution (A). Boil out H_2S filter if necessary add NH_4OH in slight excess and filter.

Ppt. B. Filtrate B.

Dissolve ppt. B in a small amount of warm HCl conc, and pour into a large beaker of water, a heavy white ppt. $= \text{BiOCl}$. Or add to a part of the HCl solution potassium stannite a black ppt. $= \text{Bi}$.

Filtrate B. If blue copper is probably present. Make a portion acid with acetic acid and add $\text{K}_4\text{Fe}(\text{CN})_6$ a red-brown ppt. $= \text{Cu}_2\text{Fe}(\text{CN})_6$.

If Cu is present, make remaining solution slightly acid with H_2SO_4 dilute and pass in H_2S in slight excess, boil, filter and pass H_2S into filtrate; a bright yellow ppt. $= \text{CdS}$.

Tin Group (Second), Sn^{II} , Sn^{IV} , As^{III} , As^{V} , Sb .

Tin forms two classes of compounds stannous Sn^{II} and stannic Sn^{IV} .

Soluble stannous compounds give with:

Fixed alkalis, a white ppt. of stannous hydroxide $\text{Sn}(\text{OH})_2$, soluble in excess forming alkali stannites, as K_2SnO_2 . The alkali carbonates also give the same ppt.

Hydrogen sulphide in slightly acid solution, a brown ppt. of SnS . Soluble in HCl conc., also in $(\text{NH}_4)_2\text{S}_2$, forming $(\text{NH}_4)_2\text{SnS}_5$ (the stannic form).

Mercuric chloride, a heavy gray ppt. of HgCl and Hg .

Metals like Fe , Zn , Al , a precipitate of metallic tin.

Soluble stannic compounds give with:

Fixed alkali hydroxides and carbonates, a heavy white ppt. of stannic acid. H_2SnO_3 , soluble in excess of the fixed alkali hydroxides.

Hydrogen sulphide in slightly acid solution, a yellow ppt. of SnS_2 , soluble in concentrated HCl , $(\text{NH}_4)_2\text{S}_2$ and aqua regia.

Conc. solutions of sodium sulphate or ammonium nitrate on boiling, a heavy white ppt. of metastannic acid, $\text{H}_{10}\text{Sn}_5\text{O}_{15}$. The latter compound is also formed by action of strong HNO_3 on tin.

Compounds heated on charcoal, oxidizing flame with $\text{Co}(\text{NO}_3)_2$ give a blue green colored mass.

Arsenic.

Arsenic also forms two classes of compounds As^{III} and As^{V} .

Arsenious acid (H_3AsO_3) in solution, acid with HCl dilute gives with:

Hydrogen sulphide, a yellow flocculent ppt. of As_2S_3 , insoluble in HCl conc., soluble in $(\text{NH}_4)_2\text{S}_2$, in aqua regia and ammonium carbonate (distinction from Sb and Sn).

Thiosulphates when boiled, a yellow ppt. of As_2S_3 (distinction from Sb and Sn).

Metallic copper on boiling, a gray ppt. on the copper, of arsenic. (Reinsch.)

Marsh test, see under arsenic for both As^{III} and As^{V} .

Soluble arsenic compounds give with:

Hydrogen sulphide in acid solution, a ppt. of S and at the same time the As^{V} is reduced to the As^{III} form.

Ammonium sulphide, a soluble salt of ammonium arsenic sulphide, $(\text{NH}_4)_3\text{AsS}_4$, which when treated with HCl dilute gives a yellow ppt. of As_2S_5 .

Magnesia mixture* a white crystalline ppt. of $\text{NH}_4\text{MgAsO}_4$ (similar to that with PO_4 ions).

Ammonium molybdate on heating, of ammonium arseni-molybdate (compare with H_3PO_4).

Fleitman's Test.—Place some Al turnings in a test tube, containing KOH solution and warm, when hydrogen is evolved add some As_2O_3 or As compound cover the mouth of the test tube with a piece of filter paper moistened with AgNO_3 and allow to stand a few minutes. A black spot will appear on the paper.

Compounds of As heated on charcoal give a white vapor of As_2O_3 with an odor of garlic, the fumes are poisonous.

Antimony.

Solutions of antimony give with:

Fixed alkali hydroxides, a white ppt. of $\text{Sb}(\text{OH})_3$ soluble in excess. The same ppt. is produced by NH_4OH but is insoluble in excess. The ppt. is also soluble in tartaric acid.

Hydrogen sulphide, an orange ppt. of Sb_2S_3 , soluble in HCl conc., and in $(\text{NH}_4)_2\text{S}_2$.

Water in excess, a heavy white ppt. of the basic salt SbOCl , soluble in strong acids.

* See footnote page 35.

Nascent hydrogen, hydrogen, antimonide H_3Sb , see under Marsh test for arsenic.

Fleitman's test no reaction (distinction from arsenic).

Antimony compounds heated on charcoal, in the oxidizing flame give a white coat of Sb_2O_3 . If this is treated with a drop of $(\text{NH}_4)_2\text{S}$ it becomes orange colored due to Sb_2S_3 .

Heated in the reducing flame with Na_2CO_3 antimony compounds give a brittle metallic mass of Sb.

Gold.

Solutions of gold are precipitated by hydrogen sulphide in acid solutions Au_2S_3 . Insoluble in mineral acids, soluble in ammonium and alkali sulphides, also in aqua regia.

Potassium and sodium hydroxide give a brown ppt. of $\text{Au}(\text{OH})_3$, soluble in excess.

Ammonium hydroxide give a brown ppt. of fulminating gold.

Ferrous sulphate gives a brownish black ppt., or it may have a metallic luster, of gold.

Oxalic acid, on heating the solution, gives a bright yellow ppt. of metallic gold, the form of the ppt. depending upon conditions. Chlorine or nitric acid should be absent from the solution.

Sulphurous acid, and many other reducing agents, also precipitate gold from its solutions.

To some SnCl_2 add a drop of HNO_3 and boil $= \text{SnCl}_4$; add more SnCl_2 and pour this mixture into AuCl_3 ; a deep purple ppt. called the Purple of Cassius is formed.

Platinum.

Hydrogen sulphide in acid solution produces a black ppt. of platinic sulphide, PtS_2 . Insoluble in strong acids. Soluble in ammonium sulphide and in aqua regia.

Potassium and ammonium chloride give yellow crystalline ppts. of potassium and ammonium, platinic chloride respectively, K_2PtCl_6 ; $(NH_4)_2PtCl_6$; both insoluble in 50 per cent ethyl alcohol. Decomposed by ignition into spongy platinum.

Ferrous sulphate gives a ppt. of platinum, only after long boiling.

Oxalic acid does not give a ppt. with solutions of platinum (distinction from gold).

Stannous chloride reduces platinic to platinous compounds in strong hydrochloric acid solution. The solution becomes darker, due to the change.

Compounds of platinum heated on charcoal yield the spongy metal.

Tin Group Scheme.

Boil a portion of the clear solution with $FeSO_4$; if a black ppt. is formed add $FeSO_4$ to the rest of the solution, boil and filter.

Ppt. A. Filtrate A.

Ppt. A. Dissolve in dil. aqua regia, boil out Cl and evaporate to small bulk; Add a strong solution of NH_4Cl , a yellowish red ppt. $= (NH_4)_2PtCl_6$. Filter and test filtrate with $FeSO_4$ or oxalic acid, boil if the latter is used; a brown or yellow ppt., which on filtering, drying and burnishing, shows a yellow color $= Au$.

Filtrate A. Pour into a Marsh apparatus, and pass gases into a solution of $AgNO_3$ for several minutes, then filter.

Ppt. B. Filtrate B.

Dissolve ppt. B. in aqua regia dilute, boil out Cl, filter and add H_2S to filtrate; an orange red ppt. $= Sb_2S_3$.

Filtrate B. Add HCl dil. to ppt. Ag, filter until clear and add H_2S , a yellow ppt. $= As_2S_3$.

Mixture in the flask of the Marsh apparatus, boil until zinc is dissolved. Decant clear liquid, wash residue, once or twice, dissolve in HCl conc., filter and test filtrate with HgCl_2 ; a white or gray ppt. = HgCl , or Hg , shows the presence of Sn.

Iron Group (Third), Fe^{II} , Fe^{III} , Al. Cr, Be.

Solutions of ferrous salts give with:

Alkali hydroxides, a white ppt. rapidly becoming dark, of ferrous hydroxide, which becomes brown on standing in the air, due to oxidation to the ferric form.

Alkali carbonates, a white ppt. of ferrous carbonate, changing by oxidation to a basic ferric salt.

Hydrogen sulphide in alkaline solutions only a black ppt. of FeS , soluble in dilute mineral acids.

Alkali sulphides give a similar reaction to H_2S .

Potassium ferrocyanide, a pale blue ppt. of potassium ferrous ferrocyanide, $\text{K}_2\text{Fe}^{\text{II}}, \text{Fe}(\text{CN})_6$. Changed by exposure to air to ferric ferrocyanide, $\text{Fe}_4[\text{Fe}(\text{CN})_6]_3$ Prussian blue. Not affected by acids, decomposed by alkalies.

Potassium ferricyanide, $\text{K}_3\text{Fe}(\text{CN})_6$, a deep blue ppt. of ferrous ferricyanide, $\text{Fe}_3[\text{Fe}(\text{CN})_6]_2$, distinction from ferric salts.

Nitric acid, chlorine and other oxidizers, ferric salts.

Borax bead in the reducing flame, a light green color. If held in the oxidizing flame the iron is oxidized and the bead becomes yellow while hot, colorless when cold.

Non-magnetic compounds heated on charcoal in the reducing flame, become magnetic due to the formation of Fe_3O_4 .

Solutions of ferric salts give with:

Alkali hydroxides and also carbonates, a brown ppt. of ferric hydroxide, insoluble in excess.

Hydrogen sulphide no ppt. but are reduced to the ferrous state.

Alkali sulphide reduction of the salt to ferrous, a ppt. of ferrous sulphide FeS .

Sodium acetate on boiling, a red brown ppt. of the basic acetate $\text{Fe}(\text{OH})_x(\text{C}_2\text{H}_3\text{O}_2)_y$.

Potassium ferrocyanide, a dark blue ppt. of ferric ferrocyanide, $\text{Fe}_4[\text{Fe}(\text{CN})_6]_3$, Prussian blue.

Potassium ferricyanide, not ppt. (distinction from ferrous).

Soluble sulphocyanates, a deep red (blood red) color, distinction from ferrous.

Borax bead, oxidizing flame, yellow hot, colorless, cold.

Aluminium.

Solutions of aluminium salts give with:

Fixed alkali hydroxide, a white gelatinous ppt. of $\text{Al}(\text{OH})_3$ soluble in excess (distinction from iron).

Ammonium hydroxide, a similar ppt., insoluble in excess.

Alkaline carbonates, a white gelatinous ppt. of $\text{Al}(\text{OH})_3$, insoluble in excess (distinction from Beryllium).

Alkaline sulphides, also ppt. of the hydroxide.

Alkali phosphates, a white ppt. of AlPO_4 , insoluble in acetic, soluble in mineral acids.

Heated on charcoal the oxide remains white, but becomes blue when heated with $\text{Co}(\text{NO}_3)_2$ giving CoAl_2O_4 .

Chromium.

Soluble chromium (Cr^{III}) salts give with:

Alkali hydroxides, a green ppt. of $\text{Cr}(\text{OH})_3$ soluble in excess of fixed alkali hydroxides, insoluble in NH_4OH . The fixed alkali solution is ppted. by boiling, distinction from Al.

Hydrogen sulphide, no ppt. Acid compounds, as chro-

mates, are reduced to the basic form; the yellow color of the former changes to the blue green color of the latter.

Alkaline sulphides, similar reactions to aluminum.

Oxidizing agents by boiling or fusion the acid form, shown by change of color from blue green to yellow. The chromic acid compounds as potassium chromate, give yellow ppts. with soluble barium or lead salts in acetic acid solution.

Beryllium (Glucinum).

The compounds of this metal are similar both to aluminum and also to magnesium, in analysis, the metal is separated with the iron group.

Soluble compounds of beryllium give with:

Fixed alkali hydroxides, a white gelatinous ppt., $\text{Be}(\text{OH})_2$, soluble in excess, also soluble in ammonium carbonate distinction from the third group metals.

Ammonium hydroxide, a similar ppt.

Ammonium sulphide, a white gelatinous ppt. of $\text{Be}(\text{OH})_2$ similar to Al, distinction from Mg.

Heat on charcoal and $\text{Co}(\text{NO}_3)_2$, a gray mass, distinction from aluminum.

Iron Group Scheme.

Test a small portion for:

Ferrous iron with $\text{K}_3\text{Fe}(\text{CN})_6$, a blue ppt = Fe'' .

Ferric iron with NH_4SCN , a blood red color = Fe^{III} .

If ferrous iron is present, add 2 or 3 drops (not more) of HNO_3 conc., boil and add NH_4Cl and NH_4OH in slight excess heat to boiling and filter.

Ppt. A. Filtrate, reject.

Digest in the cold with ammonium carbonate, filter.

Residue A. Solution A.

Heat solution A to boiling, a white gelatinous ppt. = $\text{Be}(\text{OH})_2$.

Residue A, mix with some water, add Na_2O_2 , boil and filter.

Residue B = $\text{Fe}(\text{OH})_3$.

Solution B. Divide into two parts.

(1) Add NH_4Cl and boil, a white gelatinous ppt. = $\text{Al}(\text{OH})_3$.

(2) Add acetic acid and lead acetate, a heavy yellow ppt. = $\text{PbCrO}_4 = \text{Cr}$.

Zinc Group (Fourth), Zn, Mn, Co, Ni.

Solutions of zinc salts give with:

Fixed alkali hydroxides, a white ppt. of $\text{Zn}(\text{OH})_2$ soluble in excess, forming K_2ZnO_2 , potassium zincite, distinction from Mn, Ni, Co; also from copper.

Ammonium hydroxide, a similar ppt. soluble in excess.

Alkali carbonates, a white ppt. of basic carbonate, soluble in alkali hydroxides and in ammonium carbonate.

Hydrogen sulphide, an incomplete precipitation of the sulphide ZnS in neutral solutions (mineral acids set free) but completely from the acetate or in acetic acid solution, distinction from Mn, Ni, Co.

Ammonium sulphide, a white flocculent ppt. ZnS .

Sodium phosphate, a white ppt. of ZnHPO_4 , soluble in alkali hydroxides and most acids.

Salts heated on charcoal with sodium carbonate give a white coating, which on the addition of $\text{Co}(\text{NO}_3)_2$ and heated again give a green-colored mass.

Compounds heated in the borax bead are colorless, or concentrated, yellow hot, white when cold.

Manganese.

Soluble manganese salts (Mn^{II}), give with:

Fixed alkali hydroxides, a white ppt. of $\text{Mn}(\text{OH})_2$, insoluble in excess, which on exposure to air becomes brown, due to the formation of a basic manganic compound.

Ammonium hydroxide, an incomplete precipitation of the hydroxide; no ppt. is formed in the presence of NH_4Cl .

Alkali carbonates, a white ppt. of the carbonate, which oxidizes in the air, and becomes brown.

Hydrogen sulphide, no ppt. (except the acetate and this partially) not even in acetic acid solution, distinction from Zn.

Ammonium sulphide, a pink ppt. of MnS , soluble in dilute acids, distinction from Ni and Co.

Manganous salts (Mn^{II}) preferably the sulphate heated with a large volume of HNO_3 dilute and then some red lead Pb_3O_4 or PbO_2 added; after settling, show a deep purple color due to the formation of permanganic acid, HMnO_4 .

Dry salts heated on charcoal with Na_2CO_3 and some NaNO_3 , or better, fused in a nickel capsule give a green mass of sodium manganate (Mn^{VI}).

This test may also be carried out in the loop of a platinum wire; a bright green non transparent bead is formed.

Borax or microcosmic salt bead in the oxidizing flame, a violet color hot, amethyst color cold, due to manganic oxide. This bead heated in the reducing flame for some time finally becomes colorless from the reduction of the Mn_2O_3 to the colorless MnO .

The reduction of the above salt requires skill with the blowpipe and gives good practice for the continuous operation of the reducing flame.

Cobalt.

Soluble salts of cobalt give with:

Fixed alkali hydroxides, a blue ppt. of basic salt. If boiled at once, a reddish ppt. of $\text{Co}(\text{OH})_2$ insoluble in excess.

Ammonium hydroxide, also an incomplete precipitation soluble in excess.

Hydrogen sulphide in alkaline solution, or ammonium sulphide, a black ppt. of CoS , insoluble in excess, somewhat or slowly soluble in strong acids, soluble in aqua regia.

Alkali carbonates, a basic salt somewhat soluble in excess.

Alkali cyanides, a brown ppt. of $\text{Co}(\text{CN})_2$, soluble in excess, forming a double salt $\text{Co}(\text{CN})_2 \cdot 2\text{KCN}$; this treated with a few drops of HCl and then heated changes the double salt into potassium cobalticyanide, $\text{K}_3\text{Co}(\text{CN})_6$, distinction from Ni , as not reprecipitated by acids as with double cyanide of nickel.

Nitroso- β -naphthol in acetic acid solution, a dull red ppt., distinction from Ni .

Borax bead in the oxidizing flame, a blue color, hot or cold.

Heated on charcoal with the reducing flame, a paramagnetic mass.

Nickel.

Soluble salts of nickel give with:

Fixed alkali hydroxides, a light green ppt. of $\text{Ni}(\text{OH})_2$, insoluble in excess, soluble in NH_4OH .

Hydrogen sulphide in alkaline solution a black ppt of NiS , insoluble in acetic and dilute mineral acids, soluble in aqua regia.

Alkali sulphides NiS , slightly soluble in strong $(\text{NH}_4)_2\text{S}$.

Potassium cyanide, a yellow green ppt. of $\text{Ni}(\text{CN})_2$, soluble in excess, forming the double cyanide.

This solution heated with bromine water or sodium hypochlorite gives a ppt of nickelic hydroxide $\text{Ni}_2(\text{OH})_6$ (distinction from Co).

Borax bead in the oxidizing flame, a purple color hot, brown when cold.

Compounds heated on charcoal in the reducing flame give a dark, slightly para-magnetic mass.

Zinc Group Scheme.

Add HN_4Cl , HN_4OH , and $(\text{NH}_4)_2\text{S}$ in slight excess; or better than $(\text{NH}_4)_2\text{S}$ pass in H_2S gas and filter:

Ppt. Filtrate reject.

Treat ppt. with HCl dilute:

Residue A. Solution A.

Residue A, test with borax bead, blue = Co. Brown = Ni.

If blue, dissolve residue in aqua regia, boil out Cl, and treat with a hot solution of nitroso- β -naphthol until all Co is precipitated. Filter, pass H_2S into filtrate, or add $(\text{NH}_4)_2\text{S}$; a black ppt. = NiS , confirm with borax bead.

Solution A.

Boil out H_2S , add strong solution of NaOH filter.

Ppt. B. Filtrate B.

Dissolve ppt. B in warm H_2SO_4 dilute add HNO_3 conc. 1 c.c. and HNO_3 dilute, 10 c.c. heat to boiling and add a small portion of pure Pb_3O_4 , allow the mixture to settle, a deep purple colored solution = HMnO_4 .

Filtrate B, make slightly acid with acetic acid and add H_2S , a white flocculent ppt. = ZnS . Test ppt. on charcoal.

Calcium Group (Fifth), Ba, Cr, Ca and Mg.

Precipitated as carbonates in alkaline solution; by ammonium carbonate, with ammonium chloride present; the magnesium redissolves and goes into solution.

Barium.

Solutions of barium compounds give with:

Alkali carbonates, a white flocculent ppt. of BaCO_3 , insoluble in excess.

Soluble sulphates, a heavy white ppt. of BaSO_4 , insoluble in all acids.

Soluble phosphates, a white flocculent ppt. of BaHPO_4 , soluble in dilute acids.

Soluble oxalates, a white crystalline ppt. of BaC_2O_4 , soluble in mineral acids, insoluble in acetic acid.

Soluble chromates or dichromates, a yellow ppt. of BaCrO_4 , soluble in HCl or HNO_3 dilute, insoluble in acetic acid.

Volatile compounds give a yellow green flame.

Strontium.

Solutions of strontium compounds give with:

Alkali carbonates, a white flocculent ppt. of SrCO_3 , insoluble in excess.

Soluble sulphates, a heavy white ppt. SrSO_4 , insoluble in acids but more soluble than BaSO_4 ,

Soluble phosphates, also oxalates, similar ppts. to barium.

Potassium chromate a yellow ppt. of SrCrO_4 , soluble in acids, including acetic. Potassium dichromate no ppt.

Volatile compounds give a red flame (see lithium).

Calcium.

Solutions of calcium compounds give with:

Alkali carbonates, a white flocculent ppt. of CaCO_3 , insoluble in excess, soluble in acids. Soluble in carbonic acid, forming $\text{Ca}(\text{HCO}_3)_2$. On boiling the later gives CaCO_3 .

Fixed caustic alkalies, in concentrated solutions, a white flocculent ppt. of $\text{Ca}(\text{OH})_2$; ammonium hydroxide no ppt.

Soluble sulphates in strong solutions, a heavy white ppt. of CaSO_4 , slightly soluble in cold, less in hot water.

Soluble phosphates, a white flocculent ppt. of CaHPO_4 , soluble in acetic and mineral acids.

Soluble oxalates, a white crystalline ppt. of CaC_2O_4 , insoluble in acetic acid, soluble in mineral acids.

Soluble chromates, no ppt.

Volatile compounds, a yellow red flame.

Magnesium.

Solutions of magnesium compounds give with:

Fixed alkali carbonates, a white flocculent ppt. of a basic salt, MgCO_3 , $\text{Mg}(\text{OH})_2$.

Potassium and sodium hydroxides, a white ppt. of $\text{Mg}(\text{OH})_2$ insoluble in excess.

Either ammonium hydroxide, or ammonium carbonate, only a slight precipitate.

Barium hydroxide, in the absence of ammonium chloride, a white ppt. of $\text{Mg}(\text{OH})_2$.

Disodium hydrogen phosphate, in the presence of ammonium hydroxide, a white crystalline ppt of NH_4MgPO_4 , insoluble in NH_4OH , soluble in dilute acids.

Oxalates no ppt. (distinction from calcium).

The compounds give no flame test.

Calcium Group Scheme.

Make the solution slightly alkaline with NH_4OH , add NH_4Cl until clear (to dissolve the Mg salt) and $(\text{NH}_4)_2\text{CO}_3$ in slight excess; filter and wash ppt.

Filtrate A.

Ppt. A. Dissolve in warm acetic acid and test a c.c. with $\text{K}_2\text{Cr}_2\text{O}_7$ solution; a yellow ppt. = BaCrO_4 . If Ba is present,

add $\text{K}_2\text{Cr}_2\text{O}_7$ to the rest of the solution in slight excess and filter:

Ppt. B, reject. Filtrate B.

Filtrate B, make slightly alkaline with NH_4OH and add $(\text{NH}_4)_2\text{CO}_3$ in excess, filter and wash ppt.

Filtrate C, reject.

Ppt. C. Dissolve in a small quantity of warm acetic acid. Test a portion with CaSO_4 solution, a white ppt. coming down slowly = SrSO_4 . If Sr is present, add $(\text{NH}_4)_2\text{SO}_4$ in excess to remainder of the solution, filter and add $(\text{NH}_4)_2\text{C}_2\text{O}_4$ to the filtrate, white cryst. ppt. = CaC_2O_4 ; make flame test.

Filtrate A.

Add $(\text{NH}_4)_2\text{SO}_4$ and $(\text{NH}_4)_2\text{C}_2\text{O}_4$ to remove traces of Ba, Ca, etc., filter and test filtrate with Na_2HPO_4 , a white cryst. ppt. = NH_4MgPO_4 .

Sodium Group (Six), K, Na, Li, NH_4 .

Potassium.

Solutions of potassium compounds give with:

Hydrogen platonic chloride, a yellow crystalline ppt. K_2PtCl_6 , somewhat soluble in water, insoluble in strong alcohol.

Tartaric acid in concentrated solution, a white crystalline ppt. of potassium hydrogen tartrate, $\text{KHC}_4\text{H}_4\text{O}_6$, somewhat soluble in water, insoluble in alcohol.

The salts heated in the clear flame impart a violet color. In the presence of sodium the color is usually obscured by the yellow; a piece of blue glass will absorb the yellow, but allow the violet to be observed.

Sodium.

Nearly all compounds of sodium are soluble in water, so precipitation tests are of little value.

The salts (especially the chloride) heated give an intense yellow flame which may be observed in the presence of most other flame coloring substances.

Lithium.

Only a few of the salts of this metal can be precipitated. The phosphate is probably the most important.

Disodium hydrogen phosphate gives a white crystalline ppt. of Li_3PO_4 . Filter, and moisten the ppt. with HCl dilute and make flame test. A carmine flame.

Ammonium.

Solutions of ammonium compounds give with:

Hydrogen paltinic chloride, a yellow crystalline ppt of $(\text{NH}_4)_2\text{PtCl}_6$, similar to the potassium compound. Both ppts. (K and NH_4), on ignition, leave a residue of spongy platinum.

Tartaric acid, a similar ppt. to that of potassium.

Ammonium salts heated with a solution of potassium, sodium or calcium hydroxide, give ammonia gas, which changes wet red litmus paper to blue.

Nessler's reagent, a yellow color or ppt., which is a very delicate test (see ammonium).

Sodium Group Scheme.

Make flame test for sodium. Test a portion of the solution for NH_4 by heating with NaOH or KOH solution. Test fumes with wet red litmus paper.

If NH_4 is present, evaporate the remainder of the solution

to dryness, ignite to remove NH_4 salt; take up residue with a small quantity of distilled water. Add Na_2HPO_4 in slight excess; a white crystalline ppt. $= \text{Li}_3\text{PO}_4$; moisten ppt. with HCl conc. and make flame test for Li, crimson flame.

Test filtrate with H_2PtCl_6 for K, a yellow crystalline ppt. $= \text{K}_2\text{PtCl}_6$, or make flame test using blue glass.

General outline for the separation of the groups of metals. Absence of phosphates, silicates, oxalates and insoluble sulphates.

Scheme Headings for all Groups.

Tests on the Original Solution.

Sodium, flame test, yellow.

Ammonium, boiling with NaOH solution and testing for with wet red litmus paper.

Ferrous iron, addition of potassium ferricyanide gives a blue ppt.

Ferric iron, addition of ammonium thiocyanate gives a blood red color.

Stannic tin, addition of ammonium nitrate or sodium sulphate and boiling gives a white ppt., which heated on charcoal with $\text{Co}(\text{NO}_3)_2$ gives a green mass $= \text{Sn}$.

Arsenic, (As^{V}), add magnesia mixture, filter white ppt. and dissolve it in HCl dilute. Add Na_2SO_3 , boil and pass in H_2S gas; a yellow ppt. $= \text{As}_2\text{S}_3 = \text{As}(\text{ic})$.

Separations.

To the clear solution, add HCl dilute in excess; a white ppt. $=$ first group, filter and treat ppt. according to first group scheme.

Pass H_2S gas into the filtrate; ppt. may be black, brown, yellow, etc. = second group, copper and tin group subdivisions.

Filter; Ppt. A. Filtrate A.

Treat ppt. A with strong $(\text{NH}_4)_2\text{S}_2$ to dissolve the tin group.

Residue B, examine according to scheme for the Cu group.

Solution B, add HCl dilute, filter off ppt. and treat according to scheme for Sn group.

Filtrate A, boil out H_2S , filter if necessary and if Fe has been found, add one or two drops of HNO_3 conc. and boil; then apply scheme for third group.

Filter off third group and examine filtrate for fourth group. Add $(\text{NH}_4)_2\text{S}$ in slight excess, filter. Ppt. C. Filtrate C.

Test ppt. C for fourth group according to scheme.

Filtrate C, boil out $(\text{NH}_4)_2\text{S}$, filter if necessary, and apply method for fifth group.

After precipitating with $(\text{NH}_4)_2\text{CO}_3$, filter.

Ppt. D. Filtrate D.

Test ppt. D according to scheme for fifth group.

Test filtrate D for Mg as in scheme.

ACIDS

Acids, for convenience, are divided into groups, in the same way as the metals, but at the most this method is very incomplete and not nearly so satisfactory as for the metals.

In practice acids are often tested for directly, where only a few are present, using characteristic tests for that purpose. If the acids occur as insoluble compounds (like BaSO_4 , etc.), they must be brought into solution before their characteristic tests can be applied.

First Group.

The acids of this group are precipitated by barium chloride, Sulphuric, sulphurous, thiosulphuric, phosphoric (ortho) boric, hydrofluoric, silicic, carbonic, chromic, arsenious, arsenic, oxalic, tartaric, citric.

A few reactions for each will be given.

Sulphuric Acid.

Soluble sulphates give with:

BaCl_2 a heavy white ppt. of BaSO_4 , insoluble in strong acid.

Lead acetate, a heavy white ppt. of PbSO_4 , insoluble in dilute acids.

Sulphates (ex. CaSO_4) with a strong solution of calcium chloride, a heavy white ppt. of CaSO_4 , slightly soluble in cold, almost insoluble in hot water.

Sulphate heated on charcoal with Na_2CO_3 yield Na_2S . This salt placed on a silver coin and moistened with water gives a black stain of Ag_2S .

Sulphurous Acid.

Soluble sulphites give with:

Barium chloride, a white ppt. of BaSO_3 , soluble in HCl dilute; solution heated with HNO_3 conc. a heavy white ppt. of BaSO_4 , insoluble in acids.

Lead acetate, a white ppt. of PbSO_3 , soluble in HNO_3 dilute.

Silver nitrate, a white ppt. of Ag_2SO_3 (soluble in HNO_3 dilute, which on boiling gives Ag).

HCl dilute SO_2 gas recognized by its odor; no ppt. of S is formed, distinction from thiosulphates.

Carbonic Acid.

A solution of CO_2 in water (H_2CO_3) or CO_2 gas gives with :

Calcium hydroxide, a white ppt. of CaCO_3 , soluble in excess of the acid (gas) forming $\text{Ca}(\text{HCO}_3)_2$. This compound is not very stable, decomposed by boiling into CaCO_3 , CO_2 , H_2O .

Soluble carbonates give with :

Barium chloride, a white ppt. of BaCO_3 soluble in dilute acids.

Silver nitrate, a white ppt. of Ag_2CO_3 .

Calcium chloride, a white ppt. of CaCO_3 .

Carbonates (ex. $\text{Na} + \text{K}$) when heated are decomposed into CO_2 and the oxide of the metal.

Silicic Acid.

Colloidal solutions of silicic acid have been prepared and these when evaporated yield the anhydride, SiO_2 ; a white powder insoluble in water and acids except HF .

Soluble silicates give with :

Barium chloride, a white gelatinous ppt. of BaSiO_3 .

Calcium chloride, a white gelatinous ppt. of CaSiO_3 .

HCl and other strong acids, a white gelatinous ppt. of H_2SiO_3 .

Silicates heated in the sodium ammonium phosphate bead, give a cloudy bead hot, clear when cold.

Hydrofluoric Acid.

It etches glass (see fluorine), forming first SiF_4 , then H_2SiF_6 .

Soluble fluorides give with :

Barium chloride, a white ppt. of BaF_2 soluble in HNO_3 or HCl , decomposed by H_2SO_4 into BaSO_4 and HF .

Calcium chloride gives a similar reaction.

Ortho Boric Acid H_3BO_3 .

Soluble borates give with :

Barium chloride or calcium chloride, a white ppt. of meta borate, $\text{Ba}(\text{BO}_2)_2$ $\text{Ca}(\text{BO}_2)_2$, respectively.

Boric acid, or borates mixed with H_2SO_4 conc. added to alcohol and the alcohol ignited, impart a green color to the flame.

A solution of boric acid changes the yellow color of turmeric paper to red or red brown, better after drying.

Ortho Phosphoric Acid.

Soluble orthophosphates give with :

Barium chloride, a white ppt. of $\text{BaH}(\text{PO}_4)$, soluble in dilute HCl , HNO_3 and acetic acid.

Calcium chloride also gives a similar ppt. CaHPO_4 , soluble in acetic acid, distinction from calcium oxalate.

With ammonium molybdate a yellow crystalline ppt. forming easily in the cold ; distinction from arsenic and silicic acids. The ppt. is insoluble in HNO_3 , soluble in NH_4OH .

Magnesia mixture ; white crystalline ppt. of NH_4MgPO_4 .

Distinction between orth, meta and pyro (see phosphorus).

Chromic Acid.

Soluble chromates give with :

Barium chloride, a yellow ppt. of BaCrO_4 , soluble in strong acids, insoluble in acetic acid.

Calcium salts no ppt. unless very concentrated, distinction between Ba and Ca.

Silver nitrate, a reddish ppt. of Ag_2CrO_4 , soluble in nitric acid and fixed alkali hydroxides.

Lead acetate, a yellow ppt. of PbCrO_4 , soluble in nitric acid, insoluble in acetic acid.

Hydrogen sulphide, and some other reducing agents, in sulphuric acid solution, a change in color from yellow (acid form of chromium) to blue green (basic form of chromium).

H_2O_2 , a change of color, yellow to blue; the latter due to perchromic acid $\text{H}_2\text{Cr}_2\text{O}_8$ (see hydrogen dioxide).

Second Group.

Precipitated by AgNO_3 in HNO_3 dilute solution: HCl , HBr , HI , HCN , HSCN , H_2S , HNO_2 , for others see reference books.

Hydrochloric Acid.

Soluble chlorides give with:

Silver nitrate, a white curdy ppt. of AgCl , insoluble in HNO_3 dilute, soluble in NH_4OH , KCN and $\text{Na}_2\text{S}_2\text{O}_3$.

Lead acetate, a white crystalline ppt. of PbCl_2 , soluble in boiling water.

Chlorides heated with MnO_2 and H_2SO_4 the yellow green gas chlorine; distinction from iodine, bromine and fluorine.

Chlorides mixed with $\text{K}_2\text{Cr}_2\text{O}_7$ and H_2SO_4 conc. yield when heated, the red gas chromyl chloride CrO_2Cl_2 . If this gas is passed into water, on the addition of barium acetate, a yellow ppt., BaCrO_4 , will be formed. I and Br give no volatile Cr compounds.

Hydrobromic Acid.

Soluble bromides give with:

Silver nitrate, a yellow white ppt. of AgBr , less soluble in

NH_4OH than the chloride, soluble in KCN , etc., insoluble in HNO_3 dilute.

Lead acetate, a white crystalline ppt. of PbBr_2 , soluble in boiling water.

Bromides mixed with MnO_2 and strong H_2SO_4 yield a red brown gas, Br , distinction from Cl and I .

Chlorine gas acting on a bromide sets Br free (see bromine).

Hydriodic Acid.

Soluble iodides give with :

Silver nitrate, a yellow white ppt. of AgI , almost insoluble in NH_4OH , soluble in KCN , etc.

The chloride, bromide and iodide of silver are reduced by the action of light and become first bluish, then black.

Lead acetate, a yellow crystalline ppt. of PbI_2 , soluble in boiling water.

Iodides mixed with MnO_2 and strong H_2SO_4 yield a violet colored vapor $= \text{I}$, distinction from Cl and Br ; the vapor also when cooled becomes solid.

Chlorine gas acting upon an iodide sets I free (see iodine).

Hydrocyanic Acid.

Soluble cyanides give with :

Silver nitrate, a white ppt. of AgCN , soluble in KCN .

Cyanides heated with sulphuric acid give an effervescence and the odor of peach blossoms.*

A few drops of $(\text{NH}_4)_2\text{S}$ and heated sulphocyanide, this made acid with HCl dilute and FeCl_3 added gives a blood red color (see ferric iron test).

* Use great care when smelling the gas, as it is one of the most poisonous substances known.

NaOH, FeSO_4 , FeCl_3 and after heating made acid with HCl dilute a blue ppt.

Thiocyanic Acid.

Soluble thiocyanates give with.

Silver nitrate, a white ppt. of AgSCN , soluble in NH_4OH .

Ferric chloride, a deep red color, which disappears on the addition of mercuric chloride.

Nitrous Acid.

Soluble nitrites give with:

Silver nitrate, a white ppt. of AgNO_2 somewhat soluble in water.

Acetic acid and starch paste iodide mixture, a blue color.

Oxalic Acid.

Soluble oxalates give with:

Barium chloride, a heavy white ppt. of BaC_2O_4 , soluble in mineral acids, insoluble in acetic acid.

Calcium chloride, a white crystalline ppt. of $\text{Ca}_2\text{C}_2\text{O}_4$, soluble in HCl or HNO_3 , insoluble in acetic acid, distinction from phosphate.

Oxalates heated with H_2SO_4 conc. are decomposed and give CO and CO_2 gases.

If calcium (or other) oxalate, which gives no reaction with acetic acid, is ignited, it changes to carbonate; then on the addition of acetic acid it effervesces.

Tartaric Acid.

Soluble tartrates give with:

Barium chloride, a white ppt. of $\text{BaC}_4\text{H}_4\text{O}_6$, soluble in acids.

Calcium chloride, a white ppt of $\text{CaC}_4\text{H}_4\text{O}_6$, soluble in acids and fixed caustic alkalis in the cold, distinction from citric acid.

Silver nitrate, a white ppt. of $\text{Ag}_2\text{C}_4\text{H}_4\text{O}_6$, soluble in NH_4OH , and on boiling the solution a ppt of Ag is formed, distinction from citrates.

Tartrates ignited with H_2SO_4 conc., give an odor like burnt sugar, distinction from citrates, also a black residue.

Calcium hydroxide, a white ppt. of $\text{CaC}_4\text{H}_4\text{O}_6$, distinction from citrates.

Citric Acid.

Soluble citrates give with :

Barium chloride in strong solution, a white ppt. of barium citrate.

Calcium chloride, a white ppt. of calcium citrate, insoluble in sodium or potassium hydroxide, distinction from tartrate.

Calcium hydroxide solution in the cold, no ppt., distinction from tartrates.

Silver nitrate, a white ppt. of silver citrate, not reduced by boiling, distinction from tartrates.

Citrates heated with H_2SO_4 conc., a carbonaceous residue but no odor of burnt sugar (see tartrates).

Third Group.

The acids of this group are not precipitated by barium chloride or by silver nitrate.

Nitric Acid.

All nitrates, except basic nitrates, are soluble in water.

See test for nitrates under nitric acid.

Nitrates treated with H_2SO_4 dilute and starch paste iodide mixture give no blue color, distinction from nitrites.

Chloric Acid.

All chlorates are soluble in water.

Chlorates warmed (*use care*) with H_2SO_4 conc. yield a yellow green gas, chlorine dioxide ClO_2 ; its odor differs from that of chlorine.

Chlorate solutions give no ppt. with AgNO_3 solution. If a chlorate is ignited, the residue dissolved in water, and AgNO_3 then added a white ppt. will be former of AgCl , insoluble in HNO_3 dilute. distinction from nitrate, which gives no ppt. after ignition, with AgNO_3 and HNO_3 dil.

Acetic Acid.

Acetates give with:

Strong H_2SO_4 and ethyl alcohol, when heated, a characteristic odor due to ethyl acetate $\text{C}_2\text{H}_5\text{C}_2\text{H}_3\text{O}_2$.

Salicylic Acid, $\text{HC}_7\text{H}_5\text{O}_3$.

Salicylates give with H_2SO_4 conc. and methyl alcohol, when heated the characteristic odor of oil of wintergreen due to methyl salicylate $\text{CH}_3\text{C}_7\text{H}_5\text{O}_3$.

Separations.

To the clear solution (or salts of the acids) add Na_2CO_3 solution in slight excess, boil and filter.

Ppt.

Bases as carbonates or hydroxides reject.

Filtrate. Sodium salts of the acids.

First group.

Make a small portion neutral with HCl dilute and test with BaCl_2 for the first group. If present look for the following acids only: H_2SO_4 , H_3PO_4 , H_2CO_3 , $\text{H}_2\text{C}_2\text{O}_4$.

Add HCl to original, if effervescence, test gas.

Odorless = CO_2 .

Odor like a bad egg, or gas blackens lead paper = H_2S .

Odor of bitter almonds = HCN .

Odor of burning sulphur, with no ppt. of $\text{S} = \text{H}_2\text{SO}_3$.

Odor of burning sulphur, white ppt. of $\text{S} = \text{H}_2\text{S}_2\text{O}_3$.

To neutral solution add BaCl_2 in slight excess filter:

Treat ppt. with HNO_3 dilute, a white residue = BaSO_4 .

Solution: add to portions:

(1) NH_4OH until slightly alkaline, then make acid with acetic acid, a white residue = BaC_2O_4 ; ignite in a porcelain dish, cool and add HCl dil., effervescence = oxalates.

(2) Add $(\text{NH}_4)_2\text{MoO}_4$, a yellow crystalline ppt. = H_3PO_4 .

If solution after boiling with Na_2CO_3 was colorless, chromic acid is absent. If yellow, make a portion neutral with acetic acid, add CaCl_2 and filter. To filtrate, add BaCl_2 , a yellow ppt. = BaCrO_4 .

Filtrate from Na_2CO_3 ppt.:

Second group.

Make a portion nearly neutral with HNO_3 dil. and add AgNO_3 , a white curdy ppt. = second group acids.

If present, make another portion of the solution acid with HCl dil. and Test:

(1) A portion for HSCN with FeCl_3 , blood red color.

(2) A portion for I and Br with Cl water and CS_2 (see under bromine and iodine).

If Br and I are absent, a white curdy ppt. with AgNO_3 = AgCl or AgCN (unless CN was absent), the latter is also decomposed by HCl conc., AgCl is not.

Tests for other acids as HNO_3 , HClO_3 , $\text{HC}_2\text{H}_3\text{O}_2$.

To test for nitrates and chlorates. If second group acids are present add Ag_2SO_4 in slight excess, filter, and add to

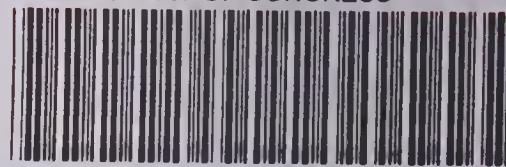
filtrate, Na_2CO_3 , filter, evaporate filtrate and ignite. After taking up with water, add AgNO_3 and HNO_3 dil., a white ppt. of $\text{AgCl} = \text{ClO}_3$.

If chlorates are present they should be removed by evaporating some of the sodium carbonate solution to dryness, and igniting carefully to drive off the oxygen from the chlorate, but not to decompose the nitrate. Take up residue with water and test for NO_3 (see Nitric Acid).

Acetic Acid.

Make a portion of the sodium carbonate solution slightly acid with H_2SO_4 dil.; add H_2SO_4 conc. ethyl alcohol and boil, the odor of ethyl acetate = acetic acid.

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